Study of Physical and Thermal Behaviour of Epoxy Composites filled with Blast Furnace Slag

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C E R T I F I C A T E

This is to certify that the work in this thesis entitled "***Study of physical and Thermal Behaviour of Epoxy Composites filled with Blast Furnace Slag"*** by **Suvendhu Kumar Patra,** has been carried out under my supervision in partial fulfillment of the requirements of the degree of **Bachelor of Technology** in *Mechanical Engineering* session of 2012 - 2013 in the department of Mechanical Engineering at National Institute of Technology, Rourkela.

To the best of my knowledge, this work has not been submitted to any other University/ Institute for the award of any degree or diploma.

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C O N T E N T S

ABSTRACT

Blast furnace slag is a major industrial waste generated in huge quantities during extraction of iron from its ores. Today eliminating, reducing or recycling waste is a major environmental remediation technique used to manage health and ecological risks. This paper aims at studying physical and thermal behaviour of epoxy composites filled with industrial waste in the form of blast furnace slag. It is already being used in concrete mixtures, soil stabilization, and metal matrix applications along with continuous research being pushed in for identifying more usage. Hence in coherence, this paper aims at exploring the possibility of BF slag's ability in manufacturing composites that can improve certain mechanical properties and becomes a possible low cost substitution to other composites. Resulting composite in this procedure has been found to have improved thermal conductivity.

Keywords: *Polymer Composites, Blast Furnace Slag, Epoxy, Thermal conductivity*

LIST of TABLES & FIGURES

CHAPTER 1

INTRODUCTION

1. INTRODUCTION

Composite Materials

Composite materials (or composites) are materials that are made from two or more structural units or constituents with largely different physical and chemical properties, which when combined produce a material with attributes different from the individual structural units and distinctive in the final structure. Of natural or synthetic ones, structural units comprise of the matrices and reinforcement structures. The binder or matrix holds the reinforcements in an orderly fashion. As the reinforcements are usually discontinuous it assists in load transfer among the reinforcements. The reinforcement adds it special mechanical and physical properties generating structures with superior properties, hence giving an upper hand over conventional monolithic materials and a large degree of freedom in designing products or even when optimizing a design. General applications have been building structures, bridges, boat hull structures, shower stalls, bathtubs, storage tanks, race car (carbon fiber bodies) etc.

Types of Composite Materials

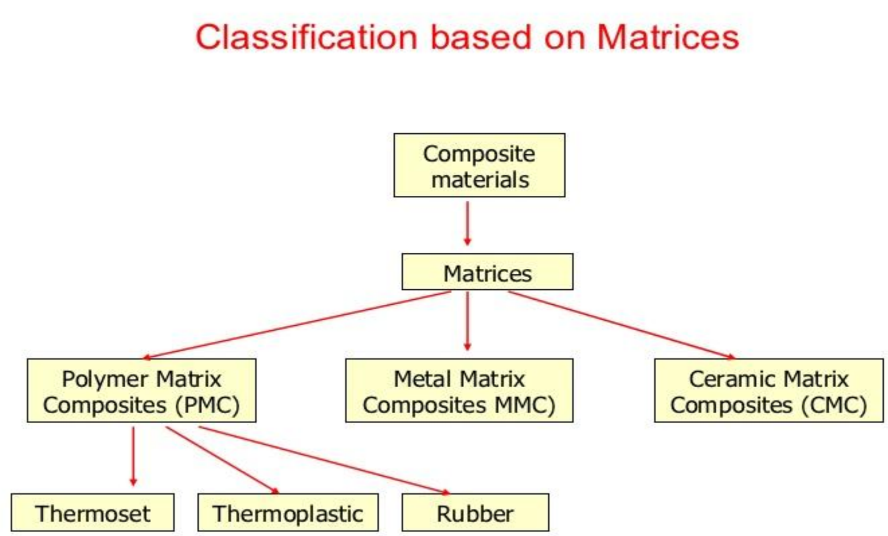

Fig 1: Classification of composites

Composites are broadly classified accordingly:

Metal Matrix Composites (MMC)

These are composites with metal being the matrix constituent, while the other can be a metal or ceramic or an organic compound. These composites are better than monolithic metals due to their high specific strength, wide range of operating temperatures, improved electrical and thermal conductivity, higher specific modulus and low co-efficient of thermal expansion etc. These attributes endorse this variety of composites for various applications such as carbide drills, tank armours, radio frequency quadruples (RFQs) etc. Modern high-performance sports cars, such as from constructors like Porsche, use rotors made of carbon fiber in a silicon carbide matrix because of its high specific heat and thermal conductivity.

Ceramic Matrix Composites (CMC)

These are composites that have ceramic fibers reinforced in ceramic matrix resulting in ceramic fiber reinforced ceramic (CFRCs) which have high fracture toughness and crack resistance. Generally it is found that there is a consequent improvement in strength and stiffness of ceramic matrix composites. The ceramic matrices are usually glass, glass ceramics (lithium aluminosilicate), carbides (SiC), nitrides (SiN4, BN), oxides (Al2O3, Zr2O3, Cr2O3, Y2O3, CaO, ThO2) and borides (ZrB2, TiB2). The reinforcements which are normally high temperature inorganic materials including ceramics may be in the form of particles, flakes, whiskers and fibers. The commonly used fibers are carbon, silicon carbide, silica and alumina. Most significant class of ceramic matrix composites is Carbon-carbon composites that can resist temperatures as high as 30000C. These consist of carbon reinforced fibers dispensed in a carbon matrix. Few applications include components for high-temperature gas turbines such as combustion chambers, stator vanes and space applications.

Polymer Matrix Composites (PMC)

These composites are also known as Fiber reinforced polymers; often use a polymer based resin as the matrix and fibers such as glass, carbon and aramid as reinforcement.

Polymers have covalently bonded macromolecular repeating structural unit, hence their mechanical property is poor when it comes to structural usage. Comparatively their strength and stiffness is low compared to metals and ceramics. Hence, this shortcoming is subdued by reinforcement of other constituent materials like ceramics or organic compounds. Low pressure and temperature require whether the polymer is a thermoset (processing temperature 200°C) or a thermoplastic (processing temperature 300°C to 400°C) along with simpler manufacturing equipment for fabrication of these composites are subject to its usage in real-life applications.

Polymer Matrix Composites can again be further classified into three groups on the basis of reinforcement material used.

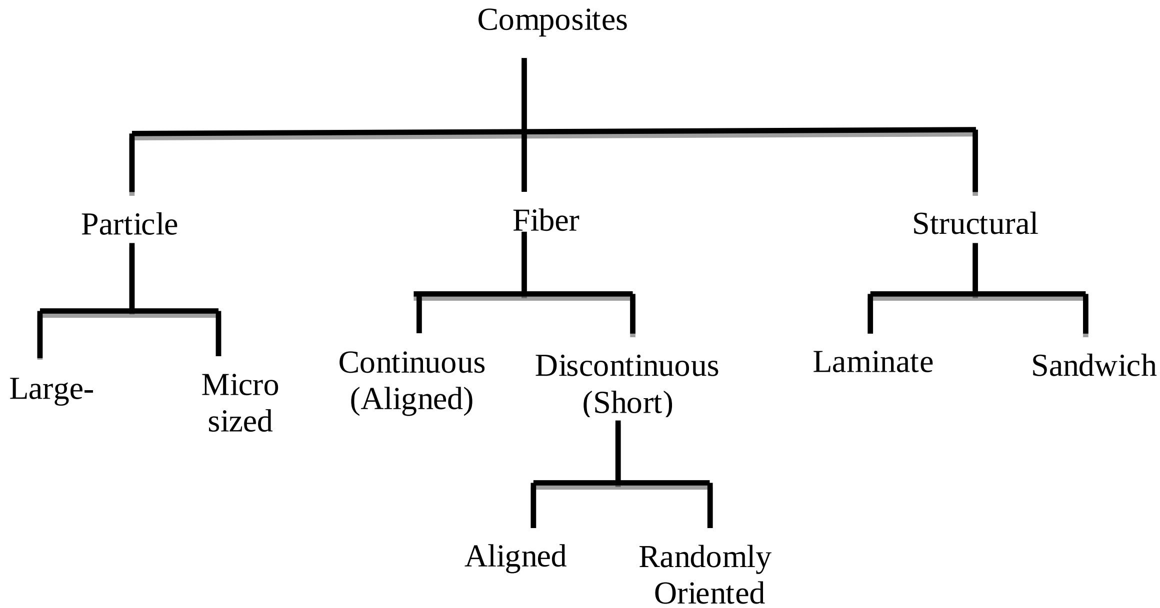

Fig 2: Classification of polymer matrix composites

BLAST FURNACE SLAG:

Background

Blast Furnace slag is a type of solid industrial waste of iron and steel making process in which when iron ore or pellets, coke and flux (limestone or dolomite) are melted together in a blast furnace. As the metallurgical smelting process is complete, the lime in the flux has chemically combines with the aluminates and silicates of the ore and coke ash to form a non-metallic product. The chemical composition of a slag varies considerably depending on the burden chemistry of the raw materials in the production process. Silicate and aluminate impurities from the ore and coke are combined in the blast furnace with a flux which reduces the viscosity of the slag. In the case of pig iron production the flux mostly contains a mixture of limestone and forsterite or in some cases dolomite. In the blast furnace the slag floats on top of the iron and is poured for separation. Slow cooling of slag melts results in an unreactive crystalline material consisting of Ca-Al-Mg silicates. To obtain a good slag reactivity, the slag melt needs to be rapidly cooled or quenched below 800 °C in order to prevent the crystallization of merwinite and melilite. To cool and fragment the slag a granulation process can be applied in which molten slag is subjected to jet streams of water or air under pressure. Alternatively, in the pelletization process the liquid slag is partially cooled with water and subsequently projected into the air by a rotating drum. In order to obtain a suitable reactivity, the obtained fragments are ground to reach the same fineness as Portland cement.

The chemical composition of BF slag for Indian conditions varies in the following range.

Calcium Oxide (CaO): 31%- 40%

Silicon Dioxide (SiO_2): 29%-38%

Aluminium Oxide (Al_2O_3): 14%-22%

Magnesium Oxide (MgO): 7%-11%

Ferrous oxide (FeO): 0.1%-1.9%

Manganese Oxide (MnO): 0.01%-1.2%

Sulphur: 1%-1.9%

Basicity (CaO/ SiO_2): 0.90%-1.3%

CHAPTER 2

LITERATURE REVIEW

2. LITERATURE REVIEW

Industrial waste today, produced from any industrial activity, major or minor, from factories, mines or mills has been a major environmental concern. Waste management and recycling processes have been major remediation techniques of the century to nullify health and ecological risks. In this context, slag happens to be a major vitreous industrial by-product of the smelting ore process. Slag generally contains metal oxides and silicon dioxide and also can contain metal sulfides and atoms in the elemental form. Major slag such as Blast furnace slag is formed when iron ore, coke and flux (either limestone or dolomite) are melted together in a blast furnace. As the smelting process is complete, the lime in the flux chemically combines with the aluminates and silicates of the ore and coke ash to form the non-metallic byproduct. While copper slag is generated of the granulated slag of copper refineries, red mud is the solid waste product of the Bayer process, the means to refine bauxite in generating alumina. These smelting processes produce large volume of non-metallic dust and soot with red mud bearing the ability to be a major environmental hazard among the three varieties of slags. However waste elimination techniques have been the savior to an extent as evident from the various solid waste reusable applications. Red mud for instance has been used in aluminium metal matrix composites for wear resistance applications, along with studies of tribological properties [1, 2] that have successfully cut part of the wastage. Red mud composite coatings [3] have improved wear resistance. Similarly synergistic effect of copper slag and red mud happen to improve physical and mechanical properties of bamboo fiber reinforced composites [4]. Again Biswas and Satpathy [5] justified the possible utilization of copper slag as filler material for the preparation of composite materials and value added products such as abrasive and cutting tools and railroad ballast. Also as copper slag is highly stable and non-leachable, it is boasted of as an alternative material for concrete applications which is evident from Sterlite Industries India's recent stake in waste management. Various copper matrix composite materials strengthened with alumina particulates are engineered by means of pressure molding and sintering. The compactness of composites increased with the sintering temperature and increase in pressure, and decreased with the alumina content increasing. The hardness of composite materials increased with the increase of sintering temperature, pressure and alumina particulates [6]. Among all slags discussed in this paper, blast furnace (BF) slag is quite the most consumed solid waste which is apparent from its numerous numbers of usages. Jian Zhou, Shunzhi Qian et al [7] studied cementitious composites from blast furnace slag and limestone powder which have high compressive strength and improved tensile strain capacity. For usage in construction purposes, Cheng and Chiu [8] researched the usage of granulated BF slag as a filler material in making of geo-polymers. Of diversified research work, remediation of chromium contaminated soils studied by O.A.B Hassan [9] claims of stabilizing chromium contaminated soils by presence of iron slag in the resulting leachate. In the present work focus has been in studying the ability of BF slag as a filler material. As this is a relatively new area of study, groundbreaking work by Padhi and Satpathy [10] and again Padhi et.al. [11] has emphasized on the erosion study of epoxy resins and hybrid epoxy composites respectively filled with blast furnace slag. In both cases, impact velocity and blast furnace slag content have been found to be major factors while dealing with the erosion study. Following work, stresses on understanding more of physical and thermal behavior of epoxy composites filled with BF slag.

CHAPTER 3

MATERIALS & METHODS

3. MATERIALS & METHODS

Matrix Material:

Epoxy LY 556 resin, chemically belongs to the epoxide family has been used as the matrix material in the present work, procured from Ciba-Geigy India Ltd. Its common name is Bisphenol-A-Diglycidyl-Ether. The low temperature curing epoxy resin (Araldite LY 556) and the corresponding hardener (HY 951) are mixed in a ratio of 10:1 by weight as recommended. Epoxy is chosen primarily because it is the most commonly used polymer and because of its insulating nature (low value of thermal conductivity, about 0.363 W/m-K) and low density (1.1 gm/cc).

Filler Material (Blast Furnace Slag):

In iron and steel making process, best practices produce around 300kg of slag/thm produced. While bad practices produce 700-800 kg of slag/thm, major practices produce 500-600 kg of slag/thm. Depending on cooling techniques, they are majorly granulated slag, air-cooled slag, pellitized or expanded slag, the smaller gradation air cooled slag or the air cooled rip rap BF slag. Density of ground granulated BF slag is in the range of 2.85-2.95 g/cm^3. Thermal conductivity of BF slag at room temperature is apparently 1.08 W/m K.

Composite Fabrication:

The low temperature curing epoxy resin (LY 556) and corresponding hardener (HY951) supplied by Ciba Geigy India Ltd. are mixed in a ratio of 10:1 by weight as needed. Blast furnace slag (avg. density of 2.85-2.95 gm/cc) with average size 100 μm is reinforced in epoxy resin (density 1.1 gm/cc) to prepare the composites. The dough (epoxy filled with blast furnace slag) is then slowly poured into the moulds, coated beforehand with wax and uniform thin film of silicone releasing agent for its excellent releasing characteristics. The composites are cast by established hand-lay-up technique so as to get disc-shaped specimens. Composites of six different compositions (of 1.4, 3.35, 5.23, 7.85, 9.4 and 11.3 vol. % of blast furnace slag respectively) are made. The castings are left to cure at room temperature for about 24 hours and then moulds are broken and samples are released to understand properties.

Table 1: Composition for mold fabrication

Samples	Composition (for blast furnace slag filled epoxy)
1	Epoxy + 1.4 vol. % (1.05 wt %) Filler
2	Epoxy + 3.35 vol. % (2.51 wt %) Filler
3	Epoxy + 5.23vol. % (3.92 wt %) Filler
4	Epoxy + 7.85 vol. % (5.88wt %) Filler
5	Epoxy + 9.4 vol. % (7.05 wt %) Filler
6	Epoxy + 11.3 vol. % (8.48 wt %) Filler

THERMAL CHARACTERIZATION

Experimental Determination of Thermal Conductivity:

Unitherm™ Model 2022 is a thermal conductivity measuring instrument for a variety of materials that includes polymers, ceramics, composites, rubbers, glasses few metals and materials of thermal conductivity from low to medium range. It requires a small test sample only. Non-solids like thin films can also be tested accurately employing a multi-layer technique. The tests concur with the **ASTM E-1530** standard.

Operating principle of Unitherm™ 2022:

The sample is held under an uniform compressive load between two polished surfaces which regulates the temperature of the two surfaces. The direction of heat flow is from the upper surface, through the sample, to the lower surface which results in an axial temperature gradient in the stack. As the thermal equilibrium is reached, the temperature difference across the sample (temperature difference between upper and lower surface) is measured along with the output from the heat flow transducer. These values and the sample thickness are the input values having been used to calculate the thermal conductivity. The temperature drop through the sample is measured with the help of temperature sensors on both side of the sample.

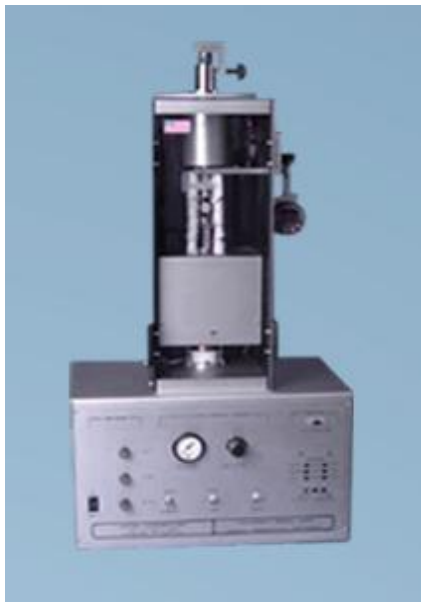

Fig 3**:** Determination of Thermal Conductivity Using Unitherm™ Model 2022

Thermal conductivity is a material property which can be described as the rate at which heat flows within a body for a given temperature difference. For one-dimensional heat flow through conduction the formula can be given as equation 3.1

$$Q= \frac{KA(T1-T2)}{x} \qquad (3.1)$$

Where Q is the heat flux (W), K is the thermal conductivity of the body (W/m-K), A is the cross sectional area (m^2), T_1-T_2 is the difference in temperature across the body (K), x is the thickness of the sample (m).

The thermal resistance of a sample can be given as:

$$R = \frac{T1 - T2}{\frac{Q}{A}} \tag{3.2}$$

Where, R is the resistance of the sample between surfaces (upper and lower) (m^2-K/W). From Equations 3.1 and 3.2 we can derive that

$$K = x/R \tag{3.3}$$

In Unitherm[tm] 2022, the heat flux transducer measures the Q value and the temperature difference is obtained between the upper and lower plate. Giving the input value of thickness of the sample and the known cross sectional area, the effective thermal conductivity of the composite samples can be calculated using Equation 3.3.

Numerical Analysis: Concept of Finite Element Method (FEM) and ANSYS:

The Finite Element Method (FEM) was introduced in 1956 by Turner et al. [12], which is a powerful computational technique for approximate solutions in a variety of engineering problems with complex domains subject to general boundary conditions. FEM has become a major step in the modeling of a physical phenomenon in various engineering fields. As the field variables vary from point to point, it results in an infinite number of solutions within the domain.

The basic concept of FEM lies in the segmentation of the domain into a finite number of sub domains (the sample in finite number of elements) for which the systematic approximate solution is constructed by applying either variational or weighted residual methods. FEM reduces the problem into a finite number of unknowns by dividing the domain into elements and expresses the unknown field variable in the form of assumed approximating functions within each element. These functions are also known as interpolation functions. These functions define the values of the field variables at specific points called nodes. Nodes connect adjacent elements. This method has the ability to differentiate the irregular domains with finite elements for which it is a valuable and practical analysis tool for the solution of boundary or initial or eigen value problems arising in various engineering fields.

Basic Steps in FEM:

The very first step is to convert the governing differential equation into an integral form.
The two techniques to achieve this are:

(i) Variational Technique

(ii) Weighted Residual Technique.

In variational technique, the integral form corresponding to the given differential equation is obtained by using calculus of variation. The solution of the problem can be obtained by the minimization of the integral. In weighted residual technique, the weighted integrals of the governing differential equation are constructed where the weight functions are known and are arbitrary except that they satisfy boundary conditions. Often this integral form is modified using the divergence theorem to reduce the continuity requirement of the solution. Then solution is obtained by setting the integral to zero.

The second step deals with the division of the domain of the problem into a number of parts, called as elements. This process of division of the domain into a finite number of elements has been known as mesh. For one-dimensional (1-D) problems, the elements are nothing but simple line segments having no shape but only length. For problems for higher dimensions, the elements have both the shape and size. For two-dimensional (2-D) or axi-symmetric problems, depending on the type of meshing the elements used are triangles, rectangles and quadrilateral having either straight or curved boundaries. For three-dimensional (3-D) problems, either tetrahedron or parallelepiped elements are used having straight or curved surfaces.

In the third step, for the interpolation functions (also called as shape functions) a proper approximation is chosen as the primary variable and the unknown values of the primary variable at some pre-selected points of the element, called as the nodes. Mostly polynomials are chosen as the shape functions. For 1-D elements, there are at least 2 nodes placed at the endpoints of the line segment. For 2-D and 3-D elements, the nodes are placed at the vertices. Additional nodes are placed on the boundaries or in the interior. Degree of freedom is the value of the primary variable at the nodes.

To get the exact solution, a complete set of polynomials (i.e., infinite term) should be in the expression for the primary variable or if it contains only the finite terms, then the number of elements will be infinite. Each of the above cases results into an infinite set of algebraic equations. Only a finite number of elements and an expression with only finite number of terms are used to make the problem tractable. The accuracy of the approximate solution, however, can be improved either by increasing the number of elements or the number of terms in the approximation.

In the fourth step, the primary variable of approximation is substituted in the integral form. It is minimized to get the algebraic equations for the unknown nodal values if the integral form is of variational type. The algebraic equations are obtained element wise first that is called the element equation and then these element equations are assembled over all the elements to get the algebraic equations for the whole domain (called as the global equation). Then the algebraic equations are modified depending on the boundary conditions and the nodal values are obtained by solving the modified algebraic equations.

Now the last step is the post-processing of the solution. Then, the nodal values are used to construct their graphical variation over the domain either in the form of graphs or contours depending on the dimensions and contours.

Advantages of the finite element method over other numerical methods are as follows:

- Any irregular-shaped domain or any type of boundary condition can be analyzed using this method. Clearly reflects the benefits.
- Analysis of domains consisting more than one material can be easily done.
- With proper refinement of the mesh or by choosing higher degree polynomials the accuracy of the solution can be improved majorly.

Analytical Model:

The effective thermal conductivity of the cube in cube model has been derived from the existing sphere in cube model proposed by J.Z. Liang and G.S. Liu. The final expression of this model is:-

$$k_{eff} = \frac{m}{\frac{m-1}{k_p} + \frac{m^2}{k_p(m^2-1)+k_f}}$$

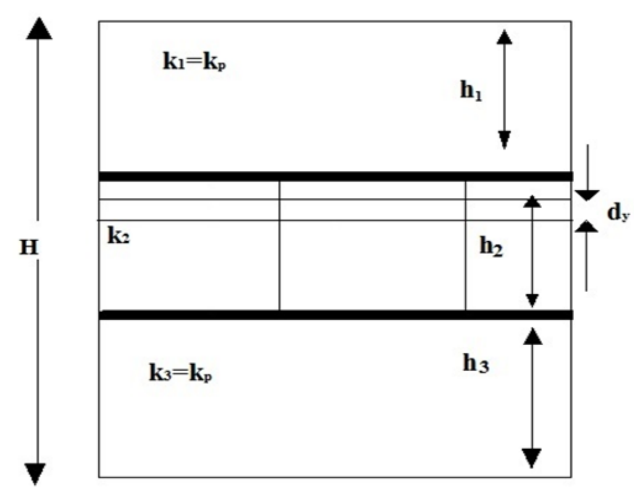

Fig 4: Cube in Cube model

Where $m = (\frac{1}{\phi f})^{1/3}$ is the volume fraction of filler material.

K_p = Thermal conductivity of polymer matrix

K_f = Thermal conductivity of filler material

K_{eff} = Effective thermal conductivity of the composite

Assumptions:

For the ideal case of thermal analysis the whole process is assumed to have following characteristics:

1. At a macroscopic level the composite is considered to be homogeneous.
2. The matrix and filler material is assumed to be completely homogeneous and isotropic.
3. Thermal contact resistance between the matrix and the filler material is assumed to be negligible or minimal.
4. Voids or material defects are not present in the composite lamina.
5. The problem considered is based on a 3D physical model.
6. The filler particles are arranged in a square-periodic array/uniformly distributed in the polymer matrix.
7. Heat conduction in the thermal model used is one-dimensional.

CHAPTER 4

RESULTS & DISCUSSION

4. RESULTS AND DISCUSSION

Numerical Analysis

In the numerical analysis of the problem, the temperatures at the nodes of the surface ABCD is prescribed as T1 (=100^0C) and the convective heat transfer coefficient is assumed to be 2.5 W/m^2-K at ambient temperature of 27°C. The heat flow direction and the boundary conditions are shown in Fig. 4.1(heat flow from face ABCD to EFGH). The other surfaces are all assumed to be adiabatic. The unknown temperatures at the nodes in the interior region and on the adiabatic boundaries are obtained with the help of finite-element program package ANSYS.

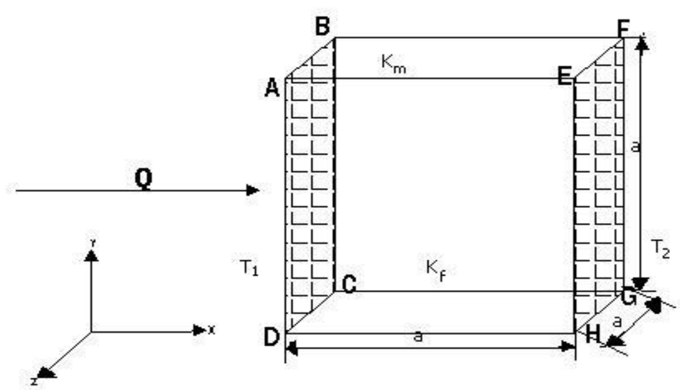

Fig.5 Boundary conditions

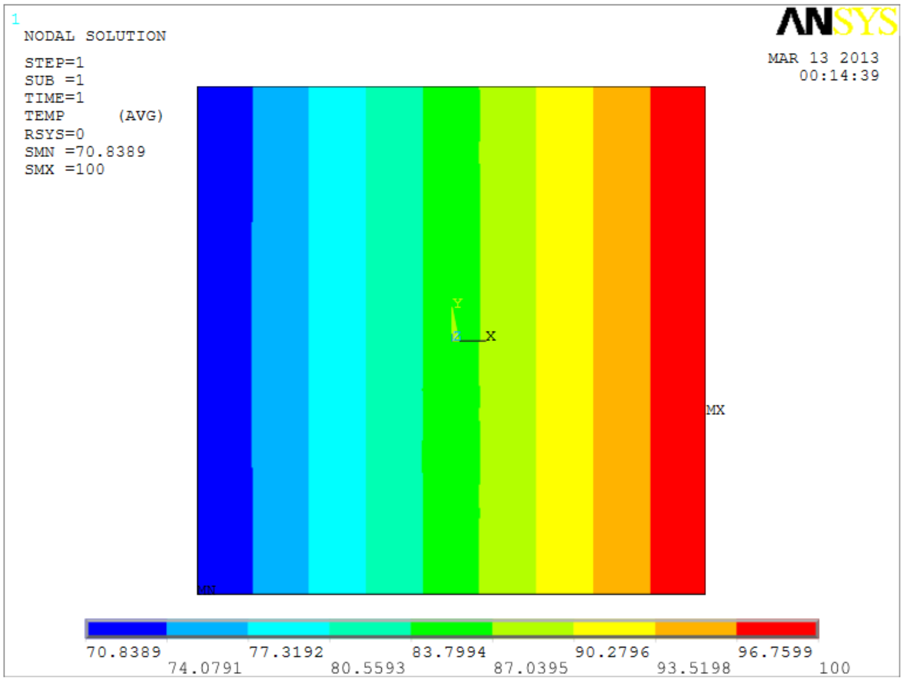

Fig 6: Temperature profile for BF slag filled epoxy composite of 1.4 % filler concentration

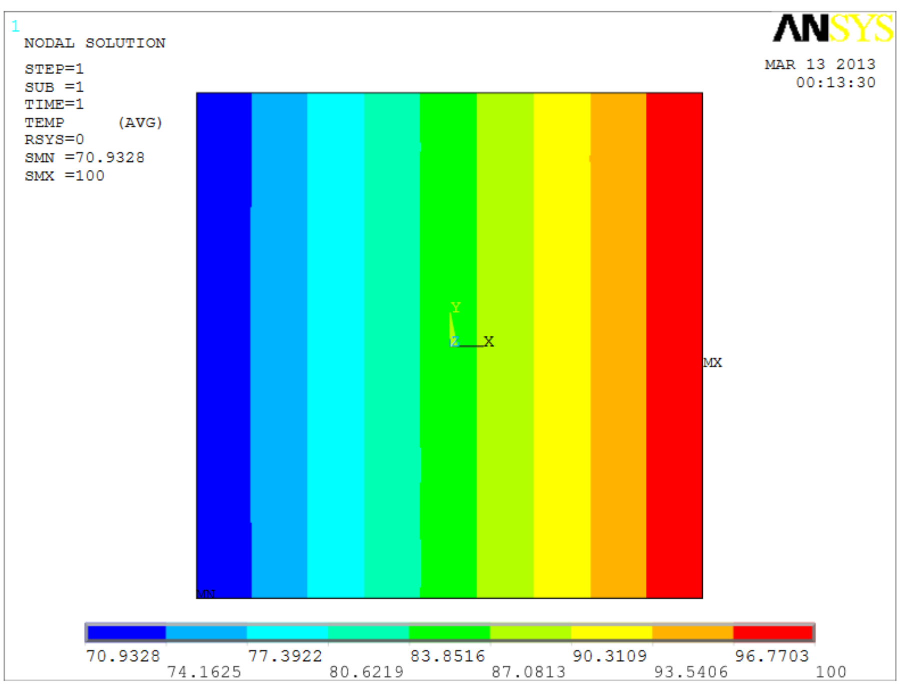

Fig 7: Temperature profile for BF slag filled epoxy composite of 3.35 % filler concentration

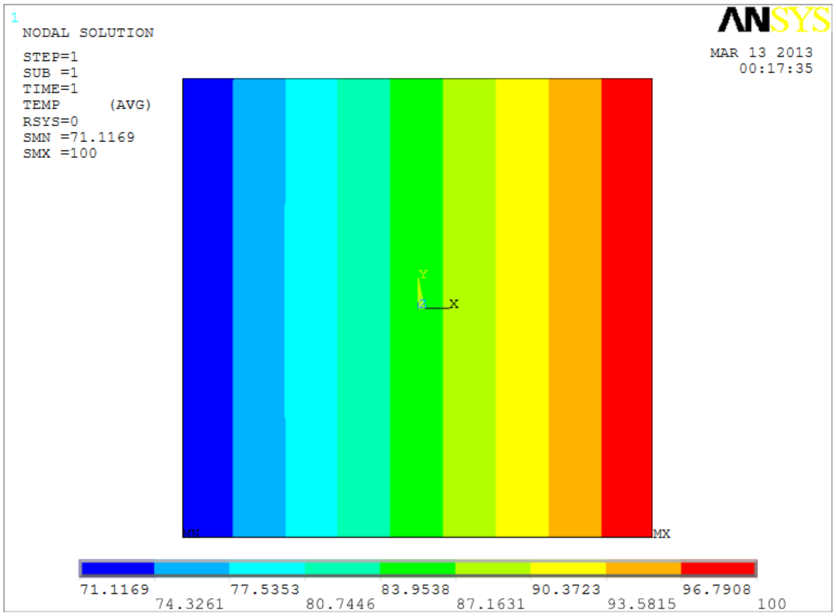

Fig 8: Temperature profile for BF slag filled epoxy composite of 5.25 % filler concentration

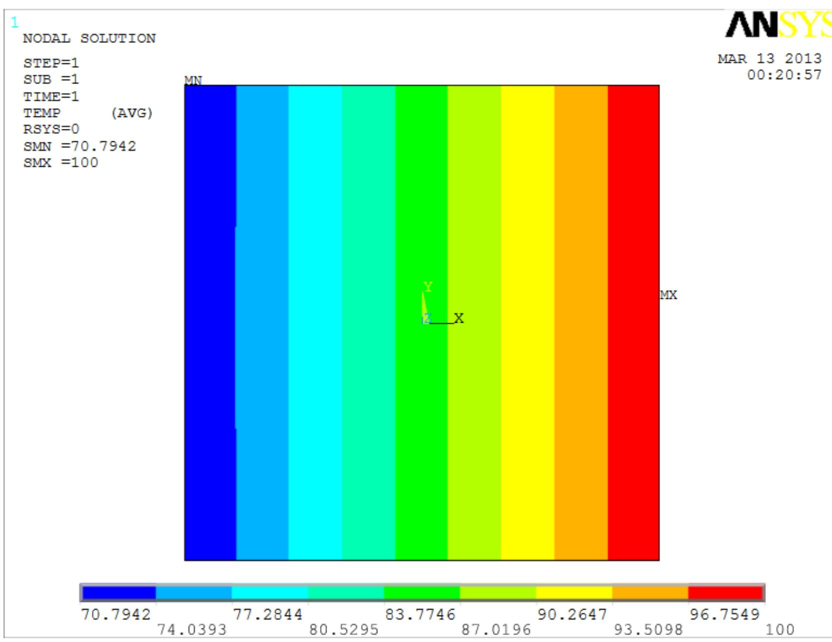

Fig 9: Temperature profile for BF slag filled epoxy composite of 7.85 % filler concentration

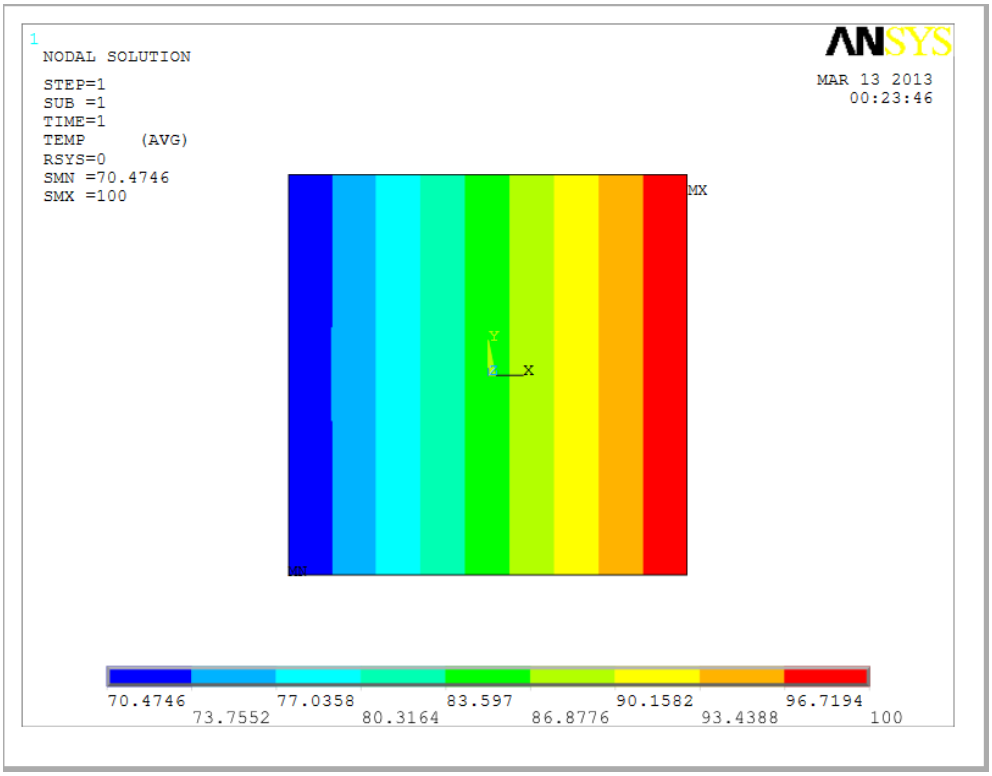

Fig 10: Temperature profile for BF slag filled epoxy composite of 9.4 % filler concentration

Table 2: Thermal conductivity for composites obtained from FEM and Experiment

Sample No	BF Slag Content Vol %	Effective Thermal Conductivity		
		FEM Simulated value (sphere in cube)	Experimental Measured Value	Analytical Model Value
1	0	0.363	0.363	0.363
2	1.4	0.37	0.378	0.465
3	3.35	0.38	0.386	0.496
4	5.23	0.40	0.412	0.515
5	7.85	0.41	0.422	0.536
6	9.4	0.42	0.425	0.547
7	11.3	0.43	0.433	0.560

Table 3: Percentage errors associated with the FEM simulated values with respect to the measured values (for blast furnace slag filled epoxy composites)

Composite Sample	BF slag Content (Vol. %)	Percentage errors associated with FEM results w.r.t. the experimental value (%)
1	1.4	2.11
2	3.35	1.55
3	5.23	2.91
4	7.85	2.84
5	9.4	1.17
6	11.3	0.69

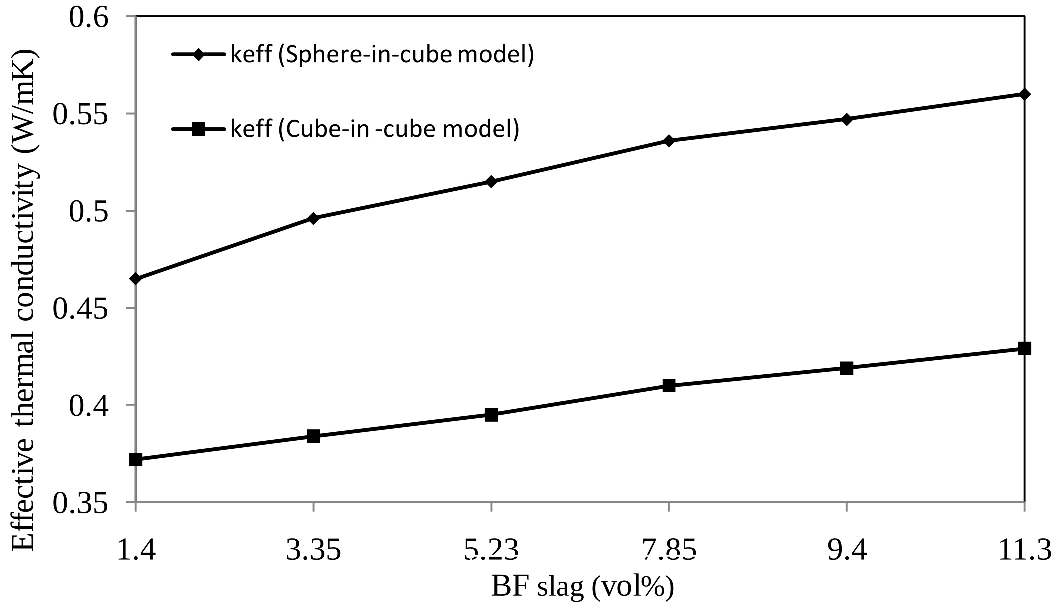

Fig.11.Comparision of effective K of Sphere-in–cube & Cube-in–cube arrangement

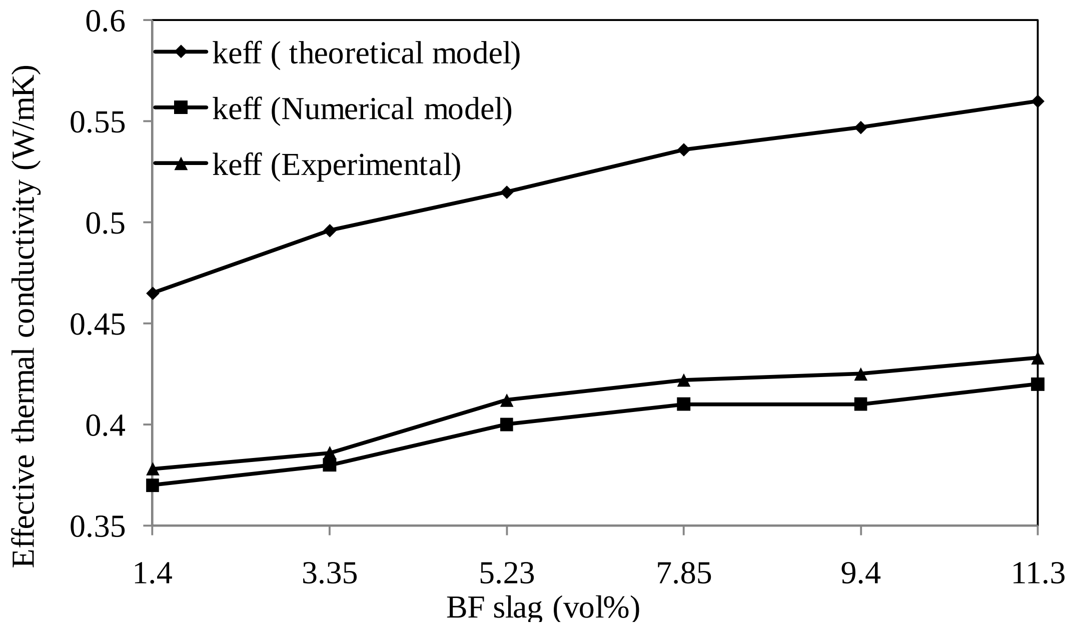

Fig.12. Comparison of numerical, theoretical and experimental values

CHAPTER 5

CONCLUSION

5. CONCLUSION

Blast Furnace Slag-filled epoxy composites can be successfully manufactured by hand-lay-up technique.

- Inspite of being an industrial waste, BF-Slag can be used as a filler material in the epoxy matrix.

- Finite Element Method (FEM) can be gainfully employed to determine the effective thermal conductivity (**keff**) of these particulate filled polymer composites for a wide range of filler concentration of BF-Slag.

- The values of the effective thermal conductivity (**keff**) obtained for various composite models from FEM are in a very well approximation with the experimental values for a wide range of filler concentration from 1.4 vol% to 11.3 vol%.

- The embedment of Blast Furnace-Slag particle results in the improvement of thermal conductivity of epoxy resin. With addition of 11.3 vol. % of filler content, the thermal conductivity improves by about 19.28% with respect to neat epoxy resin and the error percentages lie within a minimal range of 3 %

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